

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 10-053550

(43)Date of publication of application : 24.02.1998

(51)Int.Cl.

C07C 43/13  
B01J 31/08  
C07C 41/03  
// C07B 61/00

(21)Application number : 09-133282

(71)Applicant : MITSUBISHI CHEM CORP

(22)Date of filing : 23.05.1997

(72)Inventor : NAGATA KOICHI  
FURUYA TOSHIYUKI

(30)Priority

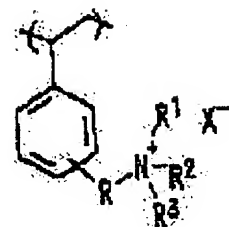
Priority number : 08134956 Priority date : 29.05.1996 Priority country : JP

## (54) PRODUCTION OF GLYCOL ETHERS

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a glycol ether of end addition structure in high activity and in high selectivity while suppressing the formation of a by-product by reacting an alkylene oxide with an alcohol in the presence of a specific anion exchange resin catalyst.

SOLUTION: An alkylene oxide such as ethylene oxide or propylene oxide is reacted with an alcohol such as a 1-6C aliphatic monohydric alcohol in the molar ratio of 2/1 to 10/1 in the presence of an anion exchange resin catalyst having a structural unit containing a quaternary ammonium group of the formula (R is an alkylene of 1-3 chain length; R1 to R3 are each a 1-4C alkyl or alkanol; X- is an anion; the benzene ring may be substituted with an alkyl or a halogen or condensed with another aromatic ring) at 50-200°C under 1-50kgf/cm<sup>2</sup> to give the objective glycol ether.



14 -

## LEGAL STATUS

[Date of request for examination]

\* NOTICES \*

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

CLAIMS

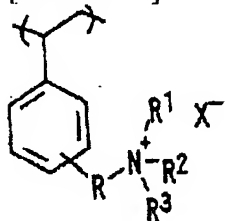
---

[Claim(s)]

[Claim 1] The manufacture approach of glycol ether that in manufacturing glycol ether by making alkylene oxide and alcohol react to the bottom of existence of a catalyst the polymer of a vinyl aromatic compound is used as a base as this catalyst, and chain length is characterized by using the anion exchange resin which has the structure where the 4th class ammonium was combined through three or more connection radicals at the aromatic series radical.

[Claim 2] The manufacture approach of glycol ether according to claim 1 that a catalyst is anion exchange resin which has the structural unit which has the 4th class ammonium expressed with the following general formula (I).

[Formula 1]

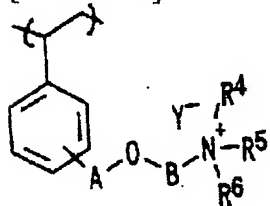


(I)

(As for R, chain length may express the alkylene group of 3-10 among a formula, and this alkylene group may contain the cyclic-hydrocarbon radical in the chain, and you may permute by the alkyl group.) R<sup>1</sup>-R<sup>3</sup> express the alkyl group or the Al Cano radical of carbon numbers 1-4, respectively, and X<sup>-</sup> expresses an anion. Moreover, the benzene ring may be permuted by the alkyl group or the halogen atom, and may be condensed with other rings.

[Claim 3] The manufacture approach of glycol ether according to claim 1 or 2 that a catalyst is anion exchange resin which has the structural unit which has the 4th class ammonium expressed with the following general formula (II).

[Formula 2]



(II)

(A expresses chain length's 1-4 alkylene group among a formula, B expresses chain length's 1-8 alkylene group, respectively, and you may permute by the alkyl group, respectively.) R<sup>4</sup>-R<sup>6</sup> express the alkyl group or alkanol radical of carbon numbers 1-4, respectively, and Y<sup>-</sup> expresses an anion. Moreover, the benzene ring may be permuted by the alkyl group or the halogen atom, and may be condensed with other rings.

[Claim 4] The manufacture approach of glycol ether given in any 1 term of claims 1-3 whose catalysts are anion exchange resin which has the structural unit guided from the structural unit and unsaturated hydrocarbon radical content cross-linking monomer which have the 4th class ammonium.

[Claim 5] The manufacture approach of glycol ether given in any 1 term of claims 1-4 whose alkylene oxide is ethylene oxide or propylene oxide.

[Claim 6] The manufacture approach of glycol ether given in any 1 term of claims 1-5 whose alcohol is the aliphatic series monohydric alcohol of carbon numbers 1-6.

[Claim 7] The manufacture approach of glycol ether given in any 1 term of claims 1-6 whose reaction temperature is 50-200 degrees C and whose reaction pressure the range of the mole ratio of alcohol and alkylene oxide is 2 / 1 - 10/1, and is 1 - 50 kgf/cm<sup>2</sup>.

\* NOTICES \*

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

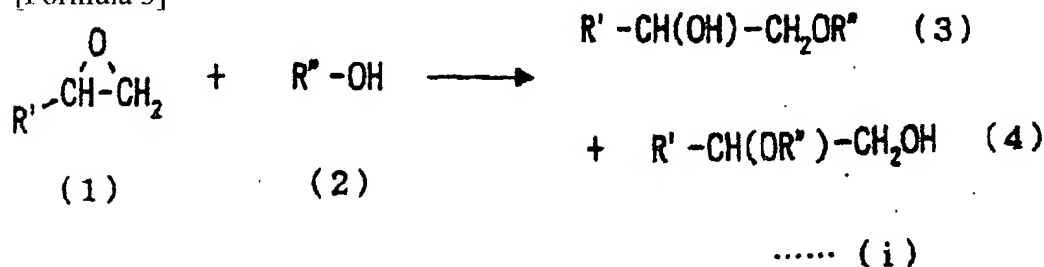
[Field of the Invention] This invention relates to the manufacture approach of glycol ether. In detail, this invention relates to the manufacture approach of the glycol ether by making alkylene oxide and alcohol react to the bottom of existence of a catalyst.

[0002]

[Description of the Prior Art] It is manufactured, when glycol ether is useful as intermediate field for manufacturing solvents, such as a coating and ink, a brake fluid combination component or glycol ether acetate, etc. and these make alkylene oxide and alcohol usually react under existence of homogeneous system base catalysts, such as an alkali-metal hydroxide and amines, (refer to the following reaction formula (i) and (ii)).

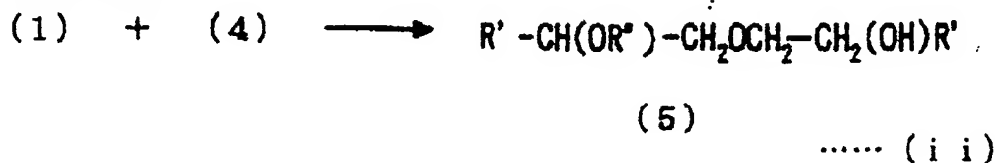
[0003]

[Formula 3]



[0004]

[Formula 4]



[0005] An end addition product (3) usually has the highest product value among each compound generated by the reaction formula (i) and (ii). However, when the above-mentioned homogeneous system base catalyst is used, there is a fault of becoming easy to generate the addition product of the form which two mols or more of alkylene oxide as shown in (5) added. Moreover, generally a homogeneous catalyst is difficult to remove from the inside of a system in a purification process. Furthermore, when unsymmetrical alkylene oxide, such as propylene oxide, is used, there is also a trouble that control of the site selectivity of a reaction is difficult and it is difficult for the end addition product (3) to wish to have to obtain preferentially.

[0006] Moreover, since the internal addition product (4) which is the position isomer of the end addition product (3) generated by the above-mentioned reaction may show toxicity, its separation and removal are needed, the yield falls, or its process increases, and it is not economically desirable.

[0007]

[Problem(s) to be Solved by the Invention] In order to solve such a trouble, some manufacture approaches of the glycol ether which makes a solid base a catalyst are proposed. For example, in Patent Publication Heisei No. 503946 [ five to ], the manufacture approach of glycol ether used considering the anionic duplex hydroxide as a catalyst is indicated. [0008] Moreover, the manufacture approach of glycol ether using the anion exchange resin containing the amino group of the second class which has the amino group of the first class, the second class, or the third class or a substituent as a catalyst, or the third class is indicated by JP,61-204142,A. When propylene oxide and ethanol are made to react in the

example, what glycol ether generated with 85% of invert ratio is described.

[0009] However, generally, since [ that heat-resistant temperature was low therefore ] the endurance was low compared with cation exchange resin, anion exchange resin had a problem in the life of a catalyst. That is, maintaining and improving the endurance of a catalyst, they are high reactivity and selectivity and the technical problem which this invention tends to solve is offering the approach the glycol ether of the structure of an end addition mold being manufactured.

[0010]

[Means for Solving the Problem] When the summary of this invention manufactures glycol ether by making alkylene oxide and alcohol react to the bottom of existence of a catalyst, the polymer of a vinyl aromatic compound is used as a base as this catalyst, and chain length consists in the manufacture approach of the glycol ether characterized by using the anion exchange resin which has the structure where the 4th class ammonium was combined through three or more connection radicals at the aromatic series radical.

[0011]

[Embodiment of the Invention] Hereafter, this invention approach is explained to a detail. In this invention, the polymer of a vinyl aromatic compound is used as a base as a catalyst, and chain length ~~uses the anion exchange resin which has the structure~~ where the 4th class ammonium was combined through three or more connection radicals for the aromatic series radical. As a monomer which constitutes the base of this anion exchange resin, styrene, vinyltoluene, ethyl vinylbenzene, vinyl naphthalene, etc. are mentioned, for example. Especially with [ chain length ] three [ or more ], the connection radical which combines the aromatic series radical and the 4th class ammonium of this base is not limited, but an alkylene group, an alkyleneoxy alkylene group, etc. are mentioned.

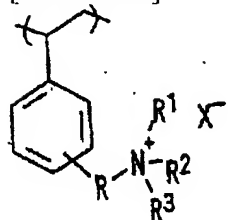
[0012] Here, chain length means the atomic number of a joining segment of the radical which connects an aromatic series radical and the 4th class ammonium. For example, if it is a straight chain alkylene group (polymethylene radical), the carbon number will serve as chain length as it is. The thing of 2 has intense degradation of resin at the time of the use in an elevated temperature [ chain length / of a connection radical ], therefore the endurance as a catalyst is inferior to it in it remarkably. Moreover, when chain length does not become [ the invert ratio of alkylene oxide ] sufficiently high, the thing of 1 and the endurance as a catalyst are inadequate.

[0013] Such anion exchange resin can be manufactured by introducing the 4th class ammonium into this permutation active group part, after usually carrying out the polymerization of the corresponding vinyl aromatic compound (henceforth a precursor monomer) of structure which has permutation active groups, such as halogen atoms, such as chlorine, a bromine, and iodine, or a tosyl group, with other monomers as occasion demands.

[0014] The anion exchange resin which has the structural unit which has the 4th class ammonium expressed with the following general formula (I) as an example of suitable anion exchange resin to use it for this invention is mentioned.

[0015]

[Formula 5]



[0016] (R may express chain length's 3-10 alkylene group among a formula, and this alkylene group may contain the cyclic-hydrocarbon radical in the chain, and you may permute by the alkyl group.) R1-R3 express the alkyl group or alkanol radical of carbon numbers 1-4, and X- expresses an anion, respectively. Moreover, the benzene ring may be permuted by the alkyl group or the halogen atom, and may be condensed with other rings.

the chain length of alkylene group R in the above-mentioned general formula (I) -- 3-10 -- it is 3-6 preferably. This alkylene group may contain the cyclic-hydrocarbon radical in the chain, and may be permuted by the alkyl group.

[0017] The example of such an alkylene group (in the following with "n-alkylene group") the straight chain alkylene group (polymethylene radical) which has a residual valence in both chain ends shall be meant, and an alkyl group shall express a methyl group or an ethyl group If it carries out, n-propylene radical, a 1-alkyl-n-propylene radical, A 2-alkyl-n-propylene radical, a 3-alkyl-n-propylene radical, n-butylene radical, a 1-alkyl-n-butylene radical, a 2-alkyl-n-butylene radical, A 3-alkyl-n-butylene radical, a 4-alkyl-n-butylene radical, n-pentene radical, A 1-alkyl-n-pentene radical, a 2-alkyl-n-pentene radical, A 3-alkyl-n-pentene radical, a 4-alkyl-n-pentene radical, A 5-alkyl-n-pentene radical, n-hexylene radical, a 1-alkyl-n-hexylene radical, A 2-alkyl-n-hexylene radical, a 3-alkyl-n-hexylene radical, A 4-alkyl-n-hexylene radical, a 5-alkyl-n-hexylene radical, A 6-alkyl-n-hexylene radical, 2, 4-dialkyl-n-pentene radical, 2, 4-dialkyl-n-hexylene radical, 2, 5-dialkyl-n-hexylene radical, 3, and 5-dialkyl-n-hexylene radical etc. is mentioned.

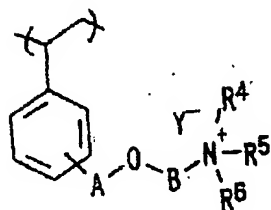
[0018] R1-R3 express the alkyl group or alkanol radical of carbon numbers 1-4, respectively, and a methyl group, an ethyl group, n-propyl group, i-propyl group, n-butyl, s-butyl, t-butyl, i-butyl, 2-hydroxyethyl radical, 3-hydroxypropyl radical, 4-hydroxy butyl, etc. are mentioned as the example.

[0019] X- expresses an anion. As an example, halogen ion, such as a hydroxide ion and fluorine ion, a chlorine ion, bromine ion, and iodine ion, etc. is mentioned. Moreover, the benzene ring may be permuted by the alkyl group or halogen atom of carbon numbers 1-4, is condensed with the ring of further others, and may form fused aromatic rings, such as a naphthalene ring and an anthracene ring.

[0020] The anion exchange resin which has the structural unit which has the 4th class ammonium expressed with the following general formula (II) as other examples of suitable anion exchange resin is mentioned.

[0021]

[Formula 6]



( I I )

[0022] (A expresses the alkylene group of carbon numbers 1-4 among a formula, B expresses the alkylene group of carbon numbers 1-8, respectively, and you may permute by the alkyl group, respectively.) R4-R6 express the alkyl group or alkanol radical of carbon numbers 1-4, respectively, and Y- expresses an anion. Moreover, the benzene ring may be permuted by the alkyl group or the halogen atom, and may be condensed with other rings.

the above-mentioned general formula (II) -- setting -- A -- the alkylene group of carbon numbers 1-4 -- B -- carbon numbers 1-8 -- the alkylene group of 4-8 is expressed preferably, respectively, and you may permute by the alkyl group, respectively. The example of such an alkylene group is the same as that of what was illustrated about alkylene group R in said general formula (I) besides a methylene group and ethylene. R4-R6 express the alkyl group or alkanol radical of carbon numbers 1-4, and they are the same as that of what was illustrated about the radicals R1-R3 in said general formula (I). [ of the example ] Moreover, anion Y- is also the same with having illustrated about X-. Moreover, the benzene ring may be similarly permuted by the alkyl group or the halogen atom, and may be condensed with other rings.

[0023] As for the anion exchange resin which has the structural unit which has the above-mentioned 4th class ammonium, it is desirable that it is the cross linked polymer. Such cross linked polymer can be manufactured by introducing the 4th class ammonium, for example, after carrying out copolymerization of said precursor monomer and the unsaturated hydrocarbon radical content cross-linking monomer with the monomer of further others as occasion demands.

[0024] As the above-mentioned unsaturated hydrocarbon radical content cross-linking monomer, a divinylbenzene, divinyl toluene, divinyl naphthalene, ethylene glycol dimethacrylate, etc. are mentioned, for example. In these, a divinylbenzene is desirable. It is indicated that thermal resistance is excellent as an anion exchanger by which the specific anion exchange resin containing the structural unit guided to JP,5-49949,A from the structural unit and unsaturated hydrocarbon radical content cross-linking monomer which have the 4th class ammonium equivalent to said general formula (I) is used for an ultrapure water manufacture process.

[0025] ~~When it used as a catalyst of the reaction which this invention persons make alkylene oxide and alcohol this anion exchange resin react, and manufactures glycol ether, the site selectivity at the time of using the outstanding alkylene oxide with it other than thermal resistance found out having the outstanding property of being good=[ high and catalytic activity and ] [ unsymmetrical ]~~ Alkylene oxide and alcohol are made to react using as a catalyst the anion exchange resin which has the above-mentioned specific 4th class ammonium, and glycol ether is made to generate in this invention approach.

[0026] As the above-mentioned alkylene oxide, although aromatic series alkylene oxide, such as aliphatic series alkylene oxide, such as ethylene oxide and propylene oxide, and styrene oxide, etc. can be used, for example, especially ethylene oxide and propylene oxide are suitable. Moreover, as the above-mentioned alcohol, although phenols, such as aliphatic series dihydric alcohol, such as aliphatic series monohydric alcohol, such as a methanol, ethanol, and propanol, ethylene glycol, and propylene glycol, or a phenol, and methyl phenol, etc. can be used, for example, especially the aliphatic series monohydric alcohol of carbon numbers 1-6 is suitable.

[0027] Any of a batch type, a semi batch type, and continuous system are sufficient as a reaction method. Also in

which method, it is important to fully contact a reaction substrate and a catalyst. Therefore, it is important to choose the suitable rate of flow for suitable stirring in continuous system again, for example in a batch type. the mole ratio of the alcohol in a raw material, and alkylene oxide -- usually -- 2 / 1 - 10/1 -- it considers as the range of 3 / 1 - 7/1 preferably. The selectivity of 1:1 adducts of alcohol and alkylene oxide falls [ the rate of alcohol ] under in this range, or there is an inclination for cooling from the system of reaction to become difficult. On the other hand, when the rate of alcohol becomes more excessive than this range, great energy will be needed in case alcohol is separated from a product at a purification process.

[0028] 50-200 degrees C of reaction temperature are usually 60-120 degrees C preferably. It is usually good 1 - 50 kgf/cm<sup>2</sup> and to make reaction pressure into the range of 2 - 20 kgf/cm<sup>2</sup> preferably.

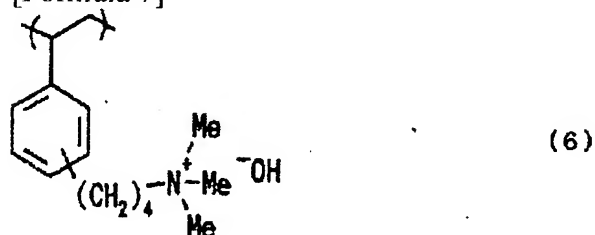
[0029]

[Example] Next, although an example explains the mode of operation of this invention concretely, this invention is not limited by the following examples unless the summary is exceeded. In addition, the "fruit 1" of front Naka, "a ratio 3", etc. show "an example 1", the "example 3 of a comparison", etc., respectively.

Propylene glycol monomethyl ether was manufactured by the reaction of propylene oxide and a methanol, using as a catalyst the anion exchange resin (it is described as "RIE-1" below) which consists of a structural unit guided from the structural unit and unsaturated hydrocarbon radical content cross-linking monomer which have the 4th class ammonium expressed with the <example 1> following type (6). In addition, the engine performance of RIE-1 is shown in Table -1.

[0030]

[Formula 7]

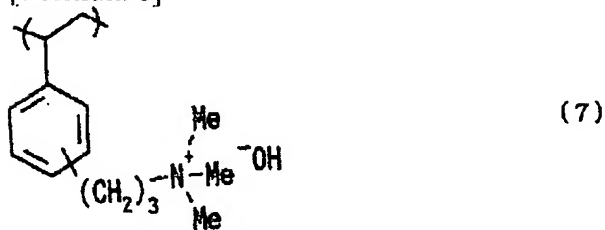


[0031] The juxtaductal type packed bed reactor was filled up with RIE-1 [ 100 cc ], and the mixed liquor (mole ratios 5/1) of a methanol and propylene oxide was passed so that the residence time (void-tower criteria) in a catalyst bed might turn into 1 hour at pressure 3.0 kgf/cm<sup>2</sup> and the temperature of 82 degrees C. As a result of analysis by the gas chromatography of effluent, the invert ratio of propylene oxide was 99%, and the methanol in a product and the percentage of 1:1 addition products of propylene oxide were 98% (1-methoxy-2-propanol the 95% of whose is an end addition product). The catalyst engine performance is maintained and change did not almost have after one-month progress in an invert ratio and selectivity. The analysis value of the effluent of one day and one month after is combined with Table -2-1, and the moisture content and exchange capacity of anion exchange resin (RIE-1) after one-month progress are combined with Table -1, and it is shown.

It reacted on the same conditions as an example 1 except having changed into the anion exchange resin (it being described as "RIE-2") which consists of a structural unit guided from the structural unit and unsaturated hydrocarbon radical content cross-linking monomer which have the 4th class ammonium expressed with the following type (7) in a <example 2> catalyst. The engine performance of RIE-2 is shown in Table -1.

[0032]

[Formula 8]



[0033] In analysis by the gas chromatography of the effluent immediately after reaction initiation, the invert ratio of propylene oxide was 98%, and the methanol in a product and the percentage of 1:1 addition products of propylene oxide were 98% (the 95% is 1-methoxy-2-propanol). The catalyst engine performance is maintained and change did not almost have after one-month progress in an invert ratio and selectivity. The analysis value of the effluent of one day and one month after is shown in Table -2-1, and the moisture content of the ion exchange resin after one-month progress (RIE-2) and exchange capacity are shown in Table -1.

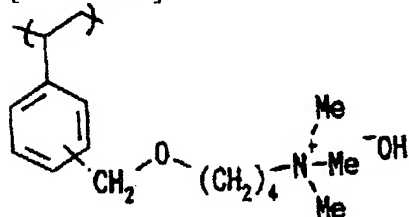
It reacted on the same conditions as an example 1 except having changed into the anion exchange resin (RIE-3) which

consists of a structural unit guided from the structural unit and unsaturated hydrocarbon radical content cross-linking monomer which have the 4th class ammonium expressed with the following type (8) in a <example 3> catalyst. The engine performance of RIE-3 is shown in Table -1.

[0034] In analysis by the gas chromatography of the effluent immediately after reaction initiation, the invert ratio of propylene oxide was 97%, and the methanol in a product and the percentage of 1:1 addition products of propylene oxide were 98% (the 95% is 1-methoxy-2-propanol). The catalyst engine performance is maintained and change did not almost have after one-month progress in an invert ratio and selectivity. The analysis value of the effluent of one day and one month after is shown in Table -2-1, and the moisture content of the ion exchange resin after one-month progress (RIE-3) and exchange capacity are shown in Table -1.

[0035]

[Formula 9]



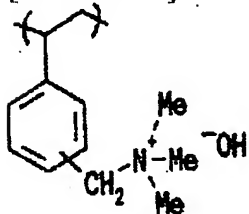
(8)

[0036] Using as a catalyst the anion-exchange-resin RIE-1 [ same ] as what was used in the <example 4> example 1, ethylene oxide and a butanol were made to react and ethylene glycol monobutyl ether was manufactured. The juxtaductal type packed bed reactor was filled up with RIE-1 [ 100 cc ], and the mixed liquor (2.6/1 of mole ratios) of a butanol and ethylene oxide was passed so that the residence time (void-tower criteria) in a catalyst bed might become in 30 minutes on pressure 15.0 kgf/cm<sup>2</sup> and conditions with a temperature of 80 degrees C. As a result of analysis by the gas chromatography of effluent, the invert ratio of ethylene oxide was 99%, and the butanol in effluent and the percentage of 1:1 addition products of ethylene oxide were 72%. The catalyst engine performance is maintained and change did not almost have after one-month progress in an invert ratio and selectivity. The analysis value of the effluent of one day and one month after is combined with Table -2-2, the moisture content of ion-exchange-resin RIE-1 after one-month progress and exchange capacity are combined with Table -1, and it is shown.

It reacted on the same conditions as an example 1 except having changed the <example 1 of comparison> catalyst into commercial anion exchange resin (the Mitsubishi Chemical make, a trade name: diamond ion SA-10A). The above "SA-10A" is anion exchange resin which has the structural unit which has the 4th class ammonium expressed with the following type (9).

[0037]

[Formula 10]



(9)

[0038] The difference in the structure of the anion exchange resin (RIE-1) and "SA-10A" which were used in the example 1 is 1 in "SA-10A" to the chain length (carbon number) of the alkylene group between the benzene ring and a nitrogen atom being 4 in RIE-1. The general property of "SA-10A" is shown in Table -1.

[0039] In analysis by the gas chromatography of the effluent immediately after reaction initiation, the invert ratio of propylene oxide was 95%, and the methanol in a product and the percentage of 1:1 addition products of propylene oxide were 98% (the 94% is 1-methoxy-2-propanol). Progress of two weeks reduced the invert ratio to 60% after reaction initiation. The analysis value of the effluent of one day and two weeks after is shown in Table -2-1, and the moisture content and exchange capacity of anion exchange resin (SA-10A) after two-week progress are shown in Table -1.

It reacted on the same conditions as an example 1 except having changed the <example 2 of comparison> catalyst into commercial anion exchange resin (the Mitsubishi Chemical make, trade name: diamond ion WA-10). The above "WA-10" is anion exchange resin which has the structural unit which has not the 4th ammonium but the 3rd class amino group. The general property of "WA-10" is shown in Table -1.

[0040] In analysis by the gas chromatography of the effluent immediately after reaction initiation, the invert ratio of propylene oxide was 90%, and the methanol in a product and the percentage of 1:1 addition products of propylene



oxide were 95% (the 94% is 1-methoxy-2-propanol). The invert ratio fell to 59% two weeks after reaction initiation. The analysis value of the effluent of one day and two weeks after is shown in Table -2-1, and the moisture content and exchange capacity of anion exchange resin (WA-10) after two-week progress are shown in Table -1.

It reacted like the example 1 except having changed the <example 3 of comparison> catalyst into the sodium hydroxide. Concentration of a catalyst was made to 100 ppm and reaction temperature was made into 100 degrees C. [0041] In analysis by the gas chromatography of the effluent immediately after reaction initiation, the invert ratio of propylene oxide was 99%, and the methanol in a product and the percentage of 1:1 addition products of propylene oxide were 93% (the 91% is 1-methoxy-2-propanol). The analysis value of early effluent is shown in Table -2-1.

The reaction of ethylene oxide and a butanol was carried out by the same reaction condition as an example 4 except having changed into the ion exchange resin (the Mitsubishi Chemical make, a trade name: diamond ion SA-10A) of the same marketing as what used the <example 4 of comparison> catalyst in the example 1 of a comparison.

[0042] In analysis by the gas chromatography of the effluent immediately after reaction initiation, the invert ratio of ethylene oxide was 96%, and the percentage of 1:1 addition products of the butanol in a product and ethylene oxide was 71%. The invert ratio fell to 80% three weeks after reaction initiation. The analysis value of the effluent of one day and three weeks after is shown in Table -2-2, and the moisture content of ion-exchange-resin SA-10A after three-week progress and exchange capacity are shown in Table -1.

The catalyst was changed into the sodium hydroxide and the reaction of the <example 5 of comparison> example 4 was carried out. Concentration of a catalyst was made to 100 ppm, reaction temperature was made into 100 degrees C, and others were made into the same conditions as an example 4, and were carried out.

[0043] In analysis by the gas chromatography of the effluent immediately after reaction initiation, the invert ratio of ethylene oxide was 99%, and the percentage of 1:1 addition products of the butanol in effluent and ethylene oxide was 61%. The analysis value of an early component is shown in Table -2-2. The data to which these experimental results were summarized are shown in Table -3.

[0044]

[Effect of the Invention] While the endurance of a catalyst improves the above-mentioned example, the example of a comparison, and by using the approach of this invention so that especially clearly from Table -3, the glycol ether of the end addition structure where product value is high can be manufactured by high activity and selectivity. Moreover, since generation of a by-product (internal addition product) is suppressed, purification cost of a product can also be made low.

[0045]

[Table 1]

<IMG SRC="/NSAPITMP2/web307/IMAGE/20070119011438228855.gif" WIDTH="448" HEIGHT="451" ALT="ID=000012">

IE capacity: (Note) The exchangeable ion number of equivalent per ion-exchange-resin unit volume [0046]

[Table 2]



[表-2-1] 反応成績 (その1)

実験 No	触媒	反応 日数	流出液組成(重量%)					AO 転化率 (%)	末端 付加 選択率 (%)
			AO	R <sup>m</sup> OH	末端 付加	内部 付加	2モル 付加		
			PO	MeOH	PM	2M1P	DPM		
実1	RIE-1	1	0.2	59.1	37.9	2.0	0.8	99	93
		30	0.2	59.2	37.8	2.0	0.8	99	93
実2	RIE-2	1	0.4	59.3	37.5	2.0	0.8	98	93
		30	0.5	59.2	37.3	2.0	0.8	98	93
実3	RIE-3	1	0.7	59.4	37.1	2.0	0.8	97	93
		30	0.7	59.3	37.2	2.0	0.8	97	93
比1	SA-10A	1	1.3	59.6	36.0	2.2	0.8	95	92
		14	10.6	64.8	22.4	1.4	0.8	60	92
比2	WA-10	1	2.6	60.6	33.0	2.0	1.8	90	89
		14	10.9	65.2	20.7	1.3	2.0	59	89
比3	NaOH	1	0.2	59.4	34.4	3.3	2.7	99	85

PO: (Note) Propylene oxide MeOH: Methanol PM:1-methoxy-2-propanol 2M1P:2-methoxy-1-propanol DPM: Dipropylene glycol monomethyl ether [0047]

[Table 3]

[表-2-2] 反応成績 (その2)

実験 No	触媒	反応 日数	流出液組成(重量%)					AO 転化率 (%)	末端 付加 選択率 (%)
			AO	R <sup>m</sup> OH	1モル 付加	2モル 付加	3モル 付加		
			EO	BuOH	EB	DEB	TEB		
実4	RIE-1	1	0.2	57.5	30.5	9.8	2.0	99	72
		30	0.2	57.4	30.5	9.8	2.0	99	72
比4	SA-10A	1	0.7	58.1	29.3	9.9	2.0	96	71
		21	3.6	62.2	24.0	8.6	1.6	80	70
比5	NaOH	1	0.2	59.8	24.4	10.4	5.2	99	61

(Note) EO: Ethylene oxide BuOH: Butanol EB: Ethylene glycol monobutyl ether DEB : Diethylene-glycol monobutyl ether TEB : Triethylene glycol monobutyl ether [0048]

[Table 4]

[表 3] 実験結果まとめ

実験 No	触媒	鎖長	初期活性(%)		触媒耐久性(残率,%)	
			AO転化率	末端付加選択率	AO転化率	IE容量
実1	RIB-1	4	99	93	100	87
実2	RIB-2	3	98	93	100	84
実3	RIB-3	6	97	93	100	73
実4	RIB-1	4	99	72	100	83
比1	SA-10A	1	95	92	63	69
比2	WA-10	--	90	89	66	66
比3	NaOH	--	99	85	----	----
比4	SA-10A	1	96	71	83	75
比5	NaOH	--	99	61	----	----

(Note) Catalyst endurance : Data \*100 of the data/first stage after the passage of time (%)

End addition selectivity: Selectivity of one-mol addition in an example 4 and the examples 4 and 5 of a comparison

[Translation done.]